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Influence of Heat Treatment on Corrosion of Mild Steel Coated with WC-Co-Al_2O_3 Cermet Composite Produced by Electrospark Deposition

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The effect of heat treatment on corrosion of C45 mild steel coated with WC-Co-Al_2O_3 cermet composite were examined. The cermet coatings (CC) were produced by electrospark deposition (ESD) technology. The content of aluminum oxide (Al_2O_3) was changed between 5 and 15 wt.%. The scanning electron microscope (SEM) was employed for the observation the surface of materials. The structure of coatings were depended from the composition of electrospark electrodes. A metallurgical bonding between the coating and substrate was obtained. Thermogravimetric measurements showed a significant effect of Al_2O_3 content on the increase of resistance to chemical corrosion of the substrate. Additionally, heat treatment of the tested coatings were made for a temperature of 400 or 800 ^{0}C. Heat treatment at 400 ^{0}C of WC-Co-Al_2O_3 cermet coatings does not affect the structure of the tested coatings. On the other hand, after heat treatment at 800 ^{0}C of WC-Co-Al_2O_3 coatings lose their anti-corrosive properties. Therefore, the chemical corrosion rate of C45 mild steel coated with WC-5Co-15Al_2O_3 coating increases more than four times compared to the material of the same specimen treated at 400 ^{0}C.

Keywords: Mild steel; WC-Co-Al_2O_3 coating; Cermet coating; Electrospark deposition; Heat treatment

1. INTRODUCTION

The electrospark deposition (ESD) process can be defined as a sort of pulsed microwelding technique that deposits an electrode material on a metallic substrate by high current/low voltage transients at short duration. Consequently, small thermal stress and few cracks are generated in ESD coatings [1,2]. Moreover, ESD is low-cost and effective method for improving the performance characteristics of metal parts. Metal coatings deposited on the metal substrates improve the corrosion resistance of the latter contributing to the improvement of the operating life of different metal parts [3-5]. The deposition of coating by means of ESD involves the use of spark discharge energy to carry the mass of the material being eroded from the carefully selected electrode (anode), which provides the coating material, to negative electrode (the cathode) which constitutes a specially modified substrate. The quality of electrospark deposition mainly depends on the shape, the duration and the average value of current or pulse power. A series of chemical and electrochemical reactions accompany ESD coating formation, which usually degrades mechanical properties and the microgeometry of applied coatings [6]. Cermet coatings as WC-Co-Al_2O_3 play a significant role in the cases where exceptional wear and erosion resistance are required [7-10]. The carbide phase imparts the coating high hardness and wear resistance, while the metal binder phase. Therefore, the CC are comparable with that of hard chrome coating, suggesting their possibility of replacement for toxic hard chrome coating. Cermet coatings produced by the electrospark deposition (ESD) technique are used for a broad range of industrial applications. As a result of ESD treatment the materials obtain greater hardness, abrasion resistance or improving the mechanical properties of the surface. In the case cermet coatings the addition of Al_2O_3 in WC-Co coating enhanced its microhardness and wear properties. The microhardness gradually increased with the increase in Al_2O_3 content as Al_2O_3 is harder than WC [11]. Moreover, the WC-12Co coating is widely adopted as an alternative to hard chrome coating, particularly for the landing gears of an aircraft, petrochemical industry and marine applications, due to environmental reasons, low production costs and equal or better material properties [12,13]. On the other hand, the limitation of WC coating is that it usually suffers from decomposition or decarburization at high temperatures [14,15]. The decarburization process induced microstructure evolution of WC coatings during the production and thermal processing is still lacking. Even though intense research effort has been devoted to understanding the decarburization mechanism of WC coating at high temperature.

The aim of the current investigations concerned the influence of heat treatment on the properties of WC-Co-Al_2O_3 cermet coatings deposited on the C45 mild steel surface by electrospark deposition (ESD) technology. The heat treatment of the tested coatings were made for a temperature of 400 or 800 ^{0}C. The rates of chemical corrosion of the materials in an atmosphere of hot air were calculated.

2. EXPERIMENTAL

2.1. Electrospark deposition

The WC-Co or WC-Co-Al_2O_3 coatings were produced by electrospark deposition. The cylindrical electrospark electrodes 5 mm in diameter and 10 mm in height were used to deposit the coatings on the C45 mild steel surface. They were produced by the impulse-plasma sintering method in a graphite matrix of tungsten carbide (particle diameter ~0,2 μm, OMG (USA)), metallic cobalt (particle diameter ~0.4 μm, Umicore (Belgium)) and aluminum oxide (particle diameter ~0.15 μm, SNI (USA)) nanopowders at the temperature of 1100 ^{0}C, under the pressure of 50 MPa. The nanopowders: WC, Co and Al_2O_3 were mixed in the appropriate proportions, and the elemental composition (wt. %) of electrospark coatings was as follows: WC-15Co-5Al_2O_3, WC-10Co-10Al_2O_3 and WC-5Co-15Al_2O_3.

An EIL-8A pulse spark generator was used to deposit coatings on the steel surface. The maximum output power of the electrospark deposition welder was 4 kW, with the voltage output of 250 V (50 Hz) continuously regulated and eight-step capacitance (470 µF). The revolving electrospark electrode was held on the deposition gun that rotates round its own axis at the speed of 240 circles/minute during the work time.

2.2. Heat treatment

Heat treatments were carried out on an NETZSCH STA Jupiter 449 thermogravimetric analyzer (TGA). A specimens were prepared in the furnace of a thermobalance under controlled temperature to obtain the corresponding thermogravimetry (TG). The TG baselines were corrected by subtraction of predetermined baselines which are determined under identical conditions except for the absence of a specimens. The specimens in the furnace were heated up from ambient temperature to 400 or 800 ^{0}C with the heating rate of 5 ^{0}C min^{-1}. In order to eliminate the effects caused by the mass and heat transfer limitations, small quantities of specimens were put into a Al_2O_3 crucible for each run under non-isothermal conditions. In addition, the oxidizing atmosphere inside the furnace of a thermogravimetric analyzer during temperature – programmed measurements was provided by means of a continuous air flow of 70 cm^3 min^{-1}. The geometric surface area of the specimen was 4.5 cm^2. Before every measurement each specimen was rinsed with double distilled water, degreased with ethanol, and was immediately immersed in the furnace of a thermobalance. The chemical corrosion rate of materials at the high temperature can be estimated by the increase in the scale thickness (X_{scale}) with time or as change mass of materials versus exposition time:

$$k_{ch} = \frac{\Delta m}{A\,t} \tag{1}$$

where Δm is the change mass of specimen, A - surface area, and t - exposition time. The chemical corrosion rate was expressed as: mg cm^{-2} h^{-1}.

2.3. Additional measuring instruments

An analysis of the phase composition of WC-Co-Al_2O_3 coating was carried out by means of the X-ray diffraction method (XRD) using a Philips PW 1830 spectrometer equipped with a copper anode tube powered with a voltage of 40 kV and a current of 30 mA, with CuKα radiation of wavelength $\lambda = 1.54$ Å. During the measurement the 2 Θ angle was changed in the range of 20 – 80^0 and the scanning velocity of 0.05^0/3 seconds.

Microhardness values of the studied specimens were determined by the Vickers (HV) method using a Microtech MX3 tester under the load of 0.4 N.

Surface topography of specimen was observed using a scanning electron microscope (SEM) Joel, type JSM-5400. The accelerating voltage was 20 kV.

3. RESULTS and DISCUSSION

3.1. Production mechanism of electrospark coating

The electrospark of coating on the steel surface was formed according to [16]:

$$W + C \rightarrow WC \qquad (2)$$

and:

$$2\,W + C \rightarrow W_2C. \qquad (3)$$

The C45 mild steel surface was covered with a heterogeneous of WC/W_2C coating which was held to the surface by metallic bonding. In conclusion of WC/W_2C coating does not protect against the oxidation of C45 mild steel surface, because numerous micro-cracks appear on the surface of coatings. Moreover, an additional layer is formed on the specimen surface as a result of metallic bonding between iron and cobalt:

$$m\,Fe + n\,Co \rightarrow Fe_mCo_n. \qquad (4)$$

The layer of Fe_mCo_n as an intermetallic bond adheres well to the C45 mild steel surface. The similar intermetallic connections of iron with different metals were observed by Deevi and Sikka [17]. Moreover, in the case of Al_2O_3 the short duration of the electrical pulse leads to the extremely rapid solidification of deposited materials and results in an exceptionally fine-grained homogeneous of WC-Co-Al_2O_3 coatings that approaches the amorphous structure.

3.2. X-ray spectra

Figure 1 shows the X-ray diffractogram (XRD) pattern of as-received WC, Co, Al_2O_3 powders and WC-Co-Al_2O_3 electrospark coatings respectively.

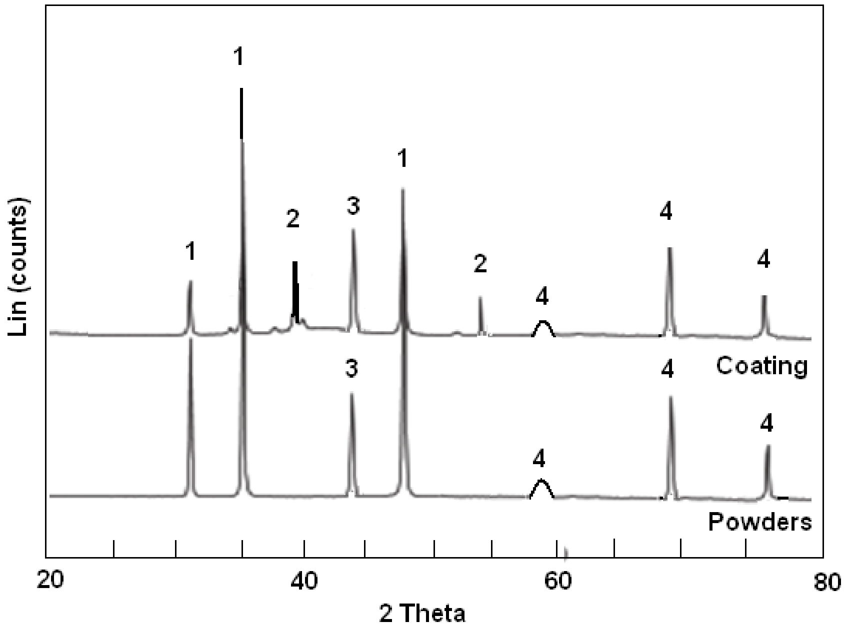

Figure 1. XRD spectra of WC, Co, Al_2O_3 powders for WC-5Co-15Al_2O_3 coating on C45 mild steel surface. The numbers were marked: 1 - WC, 2 – W_2C, 3 – Co, and 4 - Al_2O_3

The diffraction peaks observed for powders in Figure 1clearly indicate the phases corresponding to WC, Co and Al_2O_3, whereas in the WC-5Co-15Al_2O_3 cermet coating W_2C phases were also observed. The W_2C phase shows strong peak intensity, suggest slightly enhanced decarburization of WC coating. Decarburization of WC dissolved in the liquid cobalt was caused by the oxidation of C to CO or CO_2 with the precipitation of W_2C on the surface of WC crystals [18]. Moreover, for the WC-5Co-15Al_2O_3 coating (Fig. 1) in addition a phase of Al_2O_3 was appeared.

3.3. Microhardness of the materials

The microhardness values of the materials were listed in Table 1. The average microhardness of the materials were analyzed using the Vickers (HV) method.

Table 1. Average microhardness for the materials

Materials	Average microhardness HV0.4
C45 mild steel	278 ± 11
WC-15Co-5Al_2O_3	883 ± 54
WC-10Co-10Al_2O_3	938 ± 37
WC-5Co-15Al_2O_3	1042 ± 41

The microhardness HV were measured at fifteen points of the materials tested. It turned out that the average microhardness of the C45 mild steel was 278 HV0.4. When the WC-Co coatings were enriched with aluminum oxide, the average microhardness of the materials systematically increased (Table 1). However, average microhardness for the WC-5Co-15Al_2O_3 coating was about 38% higher compared to the microhardness of the substrate.

3.4. Surface topography

Figure 2 shows the SEM image of the top surface of C45 mild steel after mechanical treatment and WC-Co-Al_2O_3 cermet coatings on the steel surface. The content of Al_2O_3 was changed in the range from 5 to 15 %.

The scratches visible on the steel surface are the result of mechanical treatment (Fig. 2a)). In this case the surface of the substrate was cleaned by sand blasting treatment. The splash appearance, micro-cracks and spattering particles can be seen (Figs. 2b) – 2d)). Molten droplets formed on the electrode tip during the heating process. The droplets are accelerated by high current plasma and impinge on the substrate surface resulting in the splash in different directions. Moreover, the elemental composition (wt. %) of the all coatings is very similar to the initial composition of the electrospark electrode.

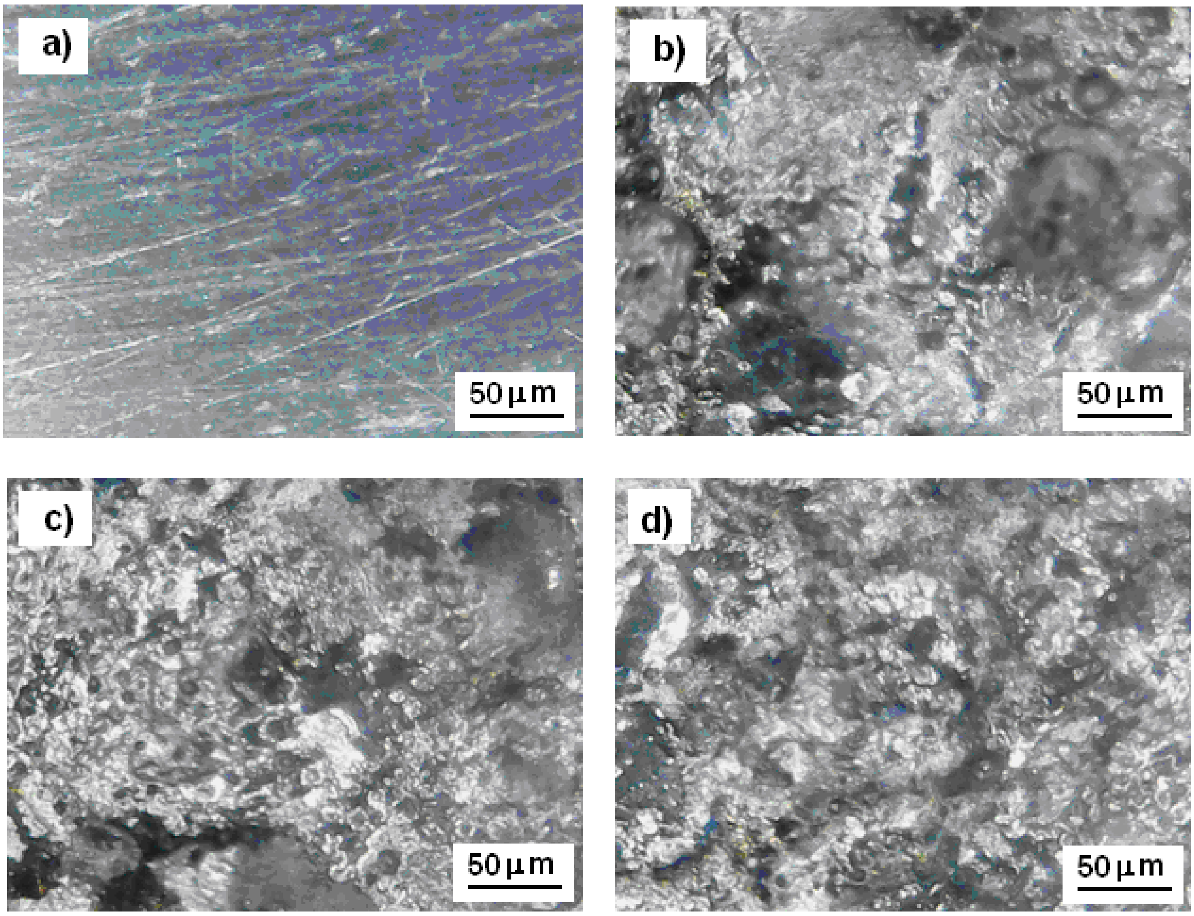

Figure 2. SEM image of the top surface: a) C45 mild steel or: b) WC-15Co-5Al_2O_3, c) WC-10Co-10Al_2O_3, and d) WC-5Co-15Al_2O_3 cermet coatings

3.5. Thermogravimetric measurements

Thermogravimetric measurements in the air atmosphere were carried out from the ambient temperature to 1200 ^{0}C. The thermogravimetric curves for C45 mild steel and WC-5Co-15Al_2O_3 coating are presented in Figure 3. However, similar thermogravimetric curves have been obtained for other WC-Co-Al_2O_3 cermet coatings.

It turned out that together with the temperature increase the mass (i.e. Δm / A) of the tested materials increases. The largest weight gain was recorded for the C45 mild steel (Fig. 3A). It has been found that after 42 hours the Δm / A for the C45 steel without and with WC-Co-Al_2O_3 cermet coating slightly increases (Fig. 3, curves (c)). It should be assumed that in high temperature conditions the surface of the tested materials was tightly covered with chemical corrosion products. The oxidation mechanism of C45 mild steel in the air atmosphere and the high temperature can be considered as electrochemical process, which we discussed in detail in the earlier article [19]. The multi-layer oxide: Fe_2O_3 / Fe_3O_4 / FeO was formed on the steel surface in the ratio: 1 / 4 / 95%. Moreover, for the WC-Co-Al_2O_3 coatings the surface was coated with cobalt oxide:

$$2\,Co + O_2 \rightarrow 2\,CoO, \qquad (5)$$

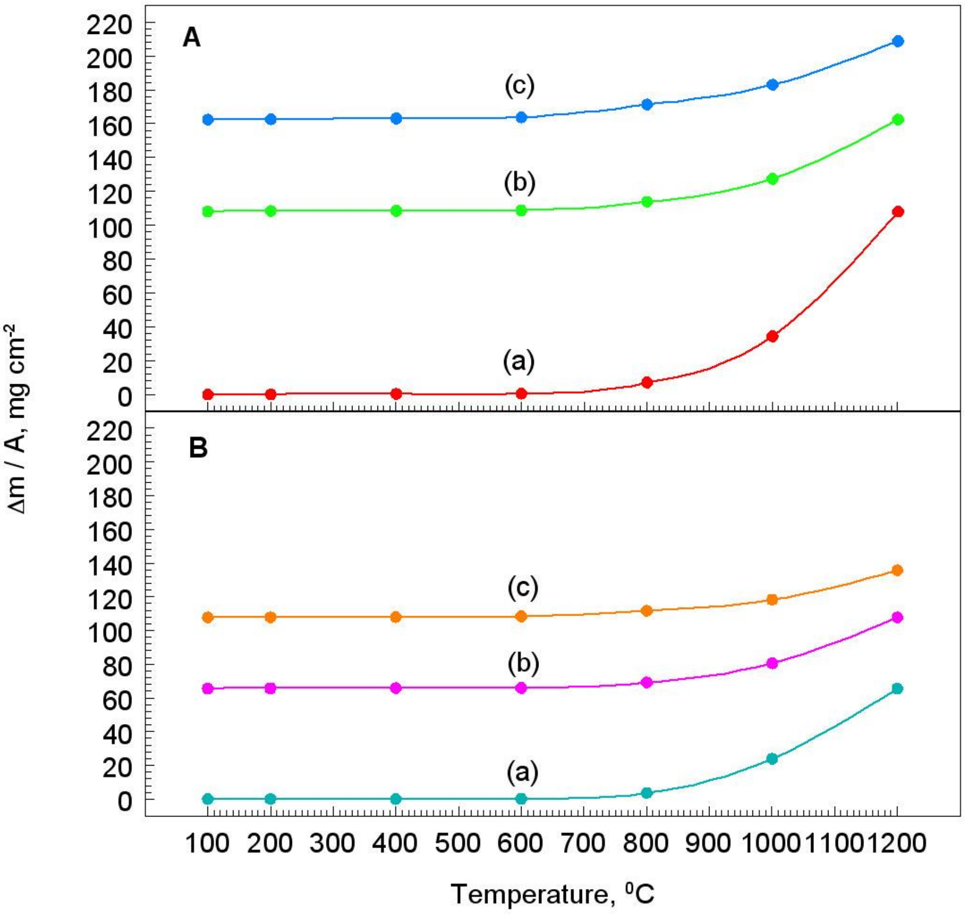

Figure 3. Thermogravimetric curves for: A C45 mild steel, B WC-5Co-15Al_2O_3 cermet coating, after exposition time: (a) 14, (b) 28, and (c) 42 hours. Heating rate of 5 ^{0}C min $^{-1}$

which additionally completes the composition of the oxide layer on the surface of the tested coatings. Therefore, the tight oxide layer protects the substrate against further oxidation. This problem will be thoroughly studied later in the work. The process of chemical corrosion occurs rapidly after exceeding the so-called critical temperature of conversion. The critical temperature of conversion for the C45 mild steel, and WC-Co-Al_2O_3 cermet coatings are shown in Figure 4.

The highest critical temperature of conversions was noted for the C45 mild steel coated with WC-5Co-15Al_2O_3 cermet coating (Fig. 4, curve (d)). Thus, the WC-5Co-15Al_2O_3 coating most effectively protects the tested steel against chemical corrosion under high temperature conditions in the air atmosphere.

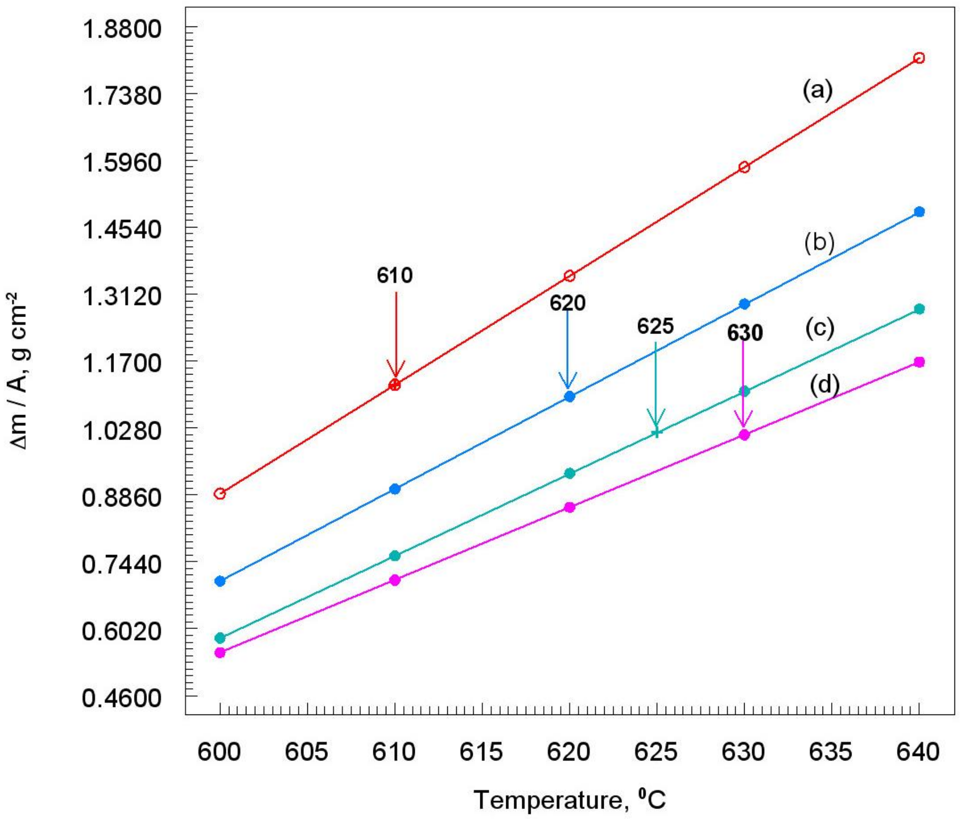

Figure 4. Critical temperature of conversion for: (a) C45 mild steel, (b) WC-15Co-5Al_2O_3, (c) WC-10Co-10Al_2O_3, and (d) WC-5Co-15Al_2O_3 cermet coatings. The critical temperature of conversion values are marked with arrows

3.6. Corrosion rate

The results of thermogravimetric measurements for the C45 mild steel in the absence and with presence of WC-Co-Al_2O_3 cermet coatings in an atmosphere of hot air were used to calculate the chemical corrosion rate (Eq. (1)) of the tested materials. The calculation results are summarized in Table 2.

Table 2. Corrosion rate of the tested materials in atmosphere of hot air

Coating	**Absence**	**WC-15Co-5Al_2O_3**	**WC-10Co-10Al_2O_3**	**WC-5Co-15Al_2O_3**
k_{ch}, mg cm^{-2} h^{-1}	1.10	0.91	0.78	0.72

The value of the chemical corrosion rate for the C45 mild steel was 1.10 mg cm^{-2} h^{-1} and for the substrate with WC-5Co-15Al_2O_3 coating was 0.72 mg cm^{-2} h^{-1} (Table 2). These results revealed that with increasing of Al_2O_3 content the corrosion rate for the tested coatings significantly decreased in the relation to the mild steel without coating (Table 2). Therefore, the WC-Co-Al_2O_3 coatings effectively protects the C45 mild steel surface against chemical corrosion process in an atmosphere of hot air [3-5]. On the other hand, the high content of Al_2O_3 in the electrospark nanopowders mixture (15%) clearly improves the anti-corrosion properties of cermet coating [11]. Therefore, the Al_2O_3 improves the tight of WC-Co layer, making difficult contact of the substrate surface with the hot air.

The oxidation kinetics in an atmosphere of hot air of C45 mild steel covered with WC-Co-Al_2O_3 cermet coatings were studied by fitting the chemical corrosion data into rate laws, Figure 5.

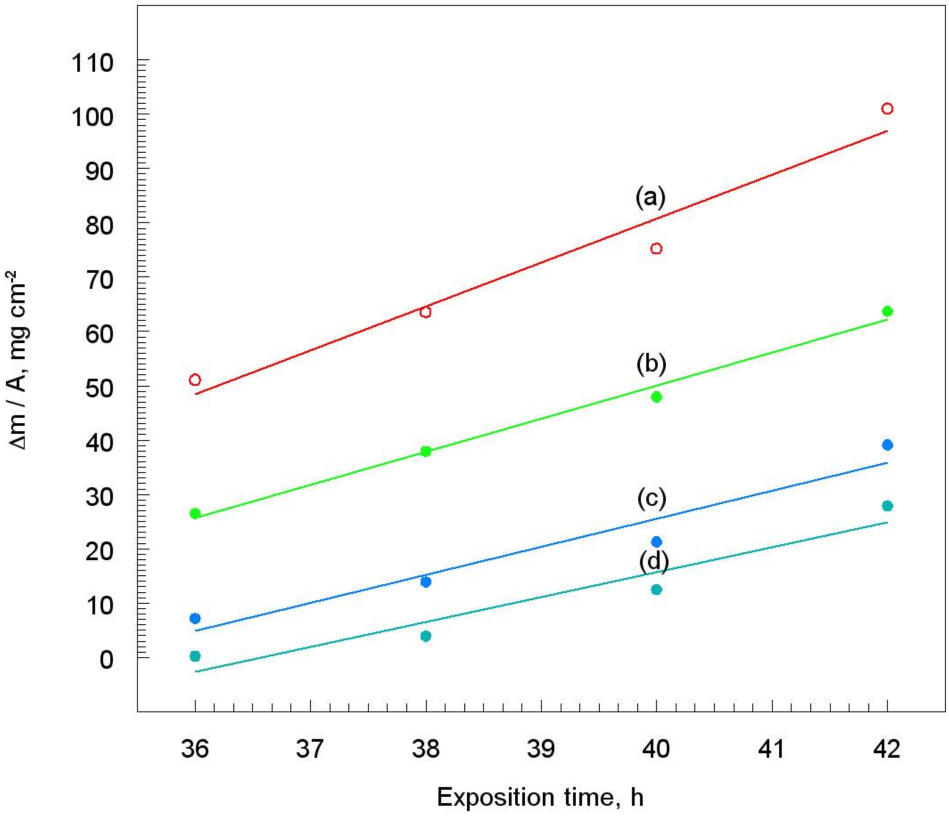

Figure 5. Change mass of materials in referring to exposition time in atmosphere of hot air for: (a) C45 mild steel, (b) WC-15Co-5Al_2O_3, (c) WC-10Co-10Al_2O_3, and (d) WC-5Co-15Al_2O_3 cermet coatings. Heating rate of 5 ^{0}C min $^{-1}$

It turned out that the oxidation kinetics of C45 mild steel covered with WC-Co-Al_2O_3 cermet coatings in atmosphere of hot air runs according to the linear function (linear law):

$$W = k_l \times t + C \qquad (6)$$

where W is the weight gain per unit area, k_l - constant oxidation rate according to linear law, t - oxidation time of the specimen, C – constant, which can be a measure of disturbances in the linear course of the oxidation process.

Oxidation according to linear law is characteristic of metals. As a result, the rate of chemical reaction is a decisive factor in the rate of metal oxidation. The linear correlation coefficients (R^2) were used to correctly match the linear equation that best describes the material oxidation process. The obtained plots were linear (Fig. 5) with good correlation coefficient ($R^2 > 0.96$), and the kinetic laws are listed in Table 3.

Table 3. Materials, linear correlation coefficients and kinetic laws of chemical corrosion

Material	R^2	**Kinetic law** (for curves (a) – (d))
C45 mild steel	0.9795	$W_a = 8.07\,t - 242$
WC-15Co-5Al$_2$O$_3$	0.9951	$W_b = 6.09\,t - 194$
WC-10Co-10Al$_2$O$_3$	0.9680	$W_c = 5.16\,t - 181$
WC-5Co-15Al$_2$O$_3$	0.9614	$W_d = 4.58\,t - 168$

Therefore, chemical corrosion of the tested materials should be classified as a zero-order reaction in atmosphere of hot air. Moreover, oxidation of C45 mild steel covered with WC-Co-Al_2O_3 cermet coatings runs according to the linear law, means that the oxide layer is cracked or porous and does not protect the substrate against chemical corrosion in high temperature conditions. On the other hand, in the case of linear law the transport of reagents to the reaction site does not encounter diffusion resistance.

3.7. Heat treatment of materials at 400 or 800 ^{0}C

The heat treatment was relied on heating at 400 or 800 ^{0}C of C45 mild steel without and with WC-Co-Al_2O_3 cermet coatings for 14 hours in an air atmosphere. The change mass of materials in referring to the exposition time are presented in Figure 6. With the increase of the heat treatment time the mass (i.e. $\Delta m / A$) of the tested materials increases. Therefore, the multi-layer oxide: Fe_2O_3 / Fe_3O_4 / FeO was formed on the C45 mild steel surface [19]. As a result of thermal treatment at 400 ^{0}C the mass of specimens of C45 mild steel covered with WC-Co-Al_2O_3 coats increased slightly (Fig. 6A). Moreover, the highest weight gain was observed for specimens subjected to heat treatment at 800 ^{0}C (Fig. 6B). Probably, in a hot air environment the compact structure of WC-Co-Al_2O_3 coatings have been destroyed [14,15].

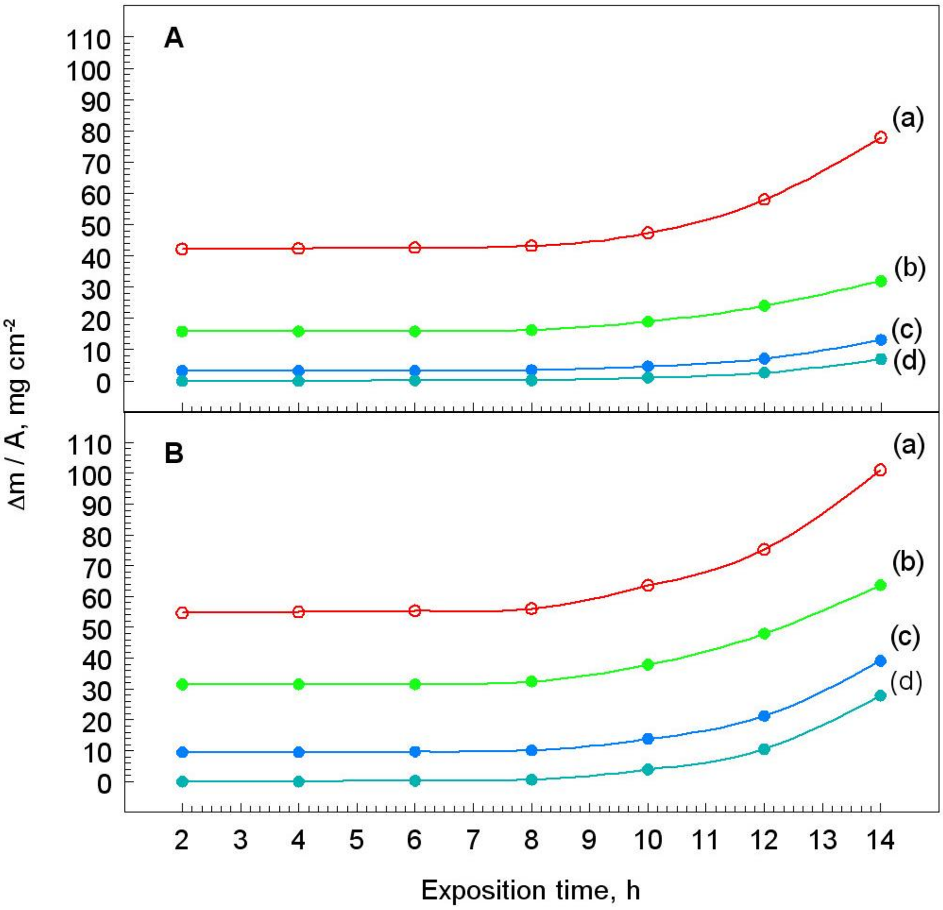

Figure 6. Change mass of materials in referring to exposition time in atmosphere of hot air at: A 400 or B 800 ^{0}C for: (a) C45 mild steel, (b) WC-15Co-5Al_2O_3, (c) WC-10Co-10Al_2O_3, and (d) WC-5Co-15Al_2O_3 cermet coatings

The results of heat treatment for the C45 mild steel in the absence and with presence of WC-Co-Al_2O_3 cermet coatings at 400 or 800 ^{0}C in the air atmosphere (Fig. 6) were used to calculate the chemical corrosion rate (Eq. (1)) of the tested materials. The calculation results are summarized in Table 4. It was found that along with the increase in the proportion of aluminum oxide in the coatings the chemical corrosion rate of the tested materials significantly decreases. However, in the case of WC-Co-Al_2O_3 coating which contains of 15% Al_2O_3 the chemical corrosion rate decreases over five times, and more than once (compared to the substrate), respectively for temperatures 400 and 800 ^{0}C. Thus, heat treatment at 400 ^{0}C of WC-Co-Al_2O_3 cermet coatings does not affect the structure of the tested coatings.

However, in the case of temperature 800 ^{0}C the tightness of coatings is destroyed, which results in free access of oxygen to the surface of the substrate. Thus, under high temperature conditions of WC-Co-Al_2O_3 cermet coatings lose their anti-corrosive properties.

Table 4. Chemical corrosion rate of tested materials at 400 or 800 ^{0}C in air atmosphere

Coating	**Absence**	**WC-15Co-5Al$_2$O$_3$**	**WC-10Co-10Al$_2$O$_3$**	**WC-5Co-15Al$_2$O$_3$**
Temperature, ^{0}C	400			
k$_{ch}$, mg cm^{-2} h^{-1}	0.57	0.26	0.16	0.11
Temperature, ^{0}C	800			
k$_{ch}$, mg cm^{-2} h^{-1}	0.74	0.51	0.47	0.44

3.7.1. Surface image

Figure 7 shows the images of the top surface of C45 mild steel without and with the WC-Co-Al_2O_3 cermet coatings after exposition 14 hours in atmosphere of air at 400 or 800 ^{0}C. Fig. 7Aa) shows of the top surface of C45 mild steel after heat treatment at 400 ^{0}C. It can be observed that the surface of mild steel was partially damaged. On the other hand, at 800 ^{0}C the substrate surface has been completely destroyed, and C45 mild steel surface was covered with a porous multilayer of oxides as: Fe_2O_3 / Fe_3O_4 / FeO. However, the oxide layer adheres well to the surface of the substrate (Fig. 7Ba). After the heat treatment of the tested materials at 400 ^{0}C the surface of WC-Co-Al_2O_3 cermet coats did not change significantly (Fig. 7Ab)-d)). What's more, the surface of the CC became homogeneous, smoother and adheres well to the substrate. Thus, it can be concluded that as a result of heat treatment at 400 ^{0}C the corrosion properties of WC-Co-Al_2O_3 cermet coatings are improved, especially for a coating that contains of 15 wt. % aluminum oxide (Fig. 7 Ad)). Therefore, the addition of Al_2O_3 to the WC-Co coating improve of anti-corrosive properties of coatings under high temperature conditions [7-11].

A completely different effect was observed during heat treatment of WC-Co-Al_2O_3 coatings at a temperature of 800 ^{0}C. In the case the destruction of WC-Co-Al_2O_3 surfaces are clearly visible (Fig. 7Bb)-d)), as a result of the interaction of the tested materials with hot air. The cobalt, which connected the coating components under these conditions was oxidized according to the reaction (5). Therefore, the surface of CC partially were covered with dark spots in the form of cobalt oxide. It should be assumed that the anti-corrosion properties of WC-Co-Al_2O_3 coatings have deteriorated significantly. The surface of the observed cermet coatings have numerous cavities, pores, and plenty cracks (Fig. 7Bb)-d)) that appeared as a result of heat treatment of tested materials at a temperature of 800 ^{0}C.

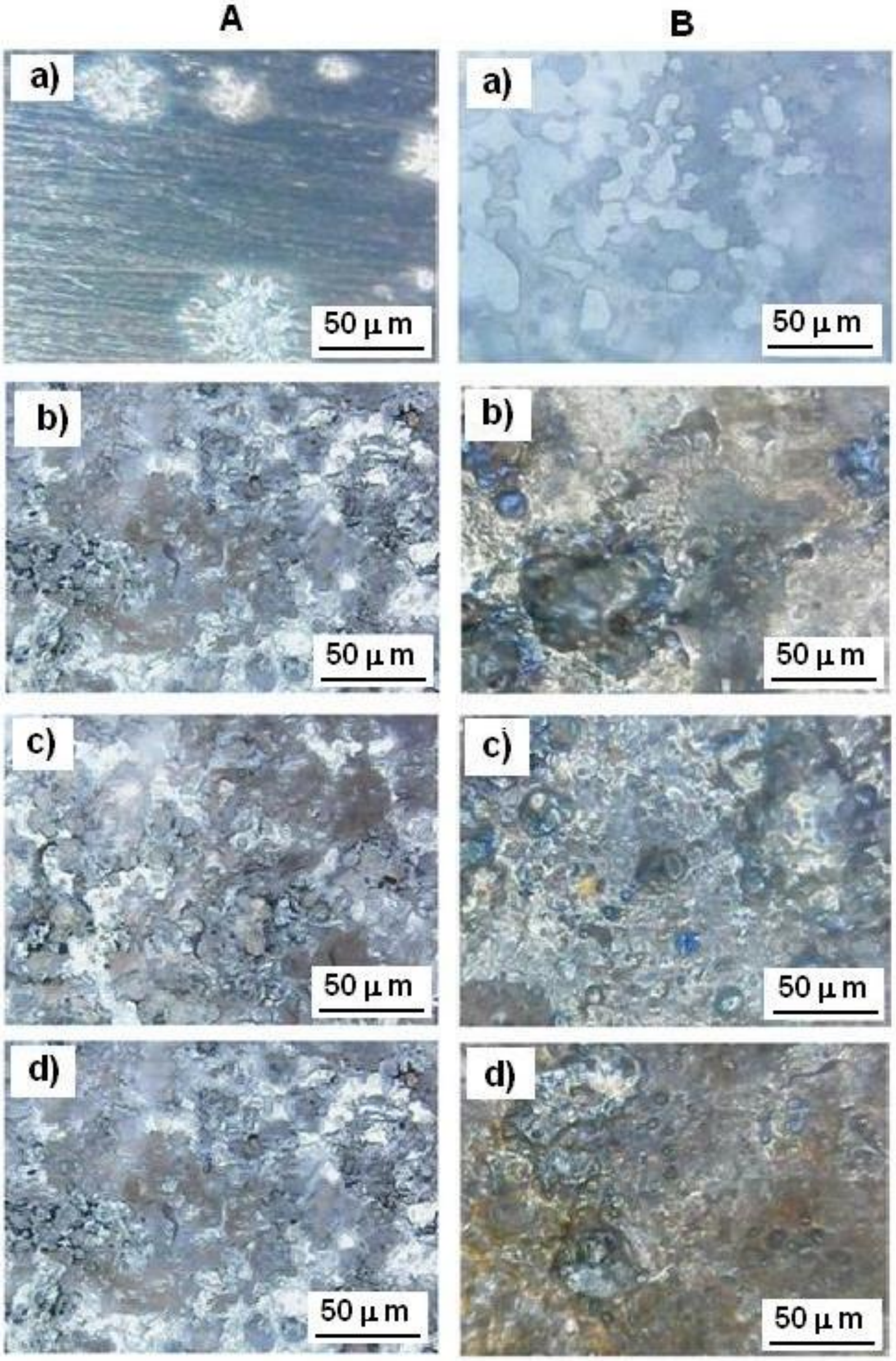

Figure 7. SEM image top surface of the tested materials after exposition 14 hours in atmosphere of air at: A 400 or B 800 ^{0}C for: (a) C45 mild steel, (b) WC-15Co-5Al_2O_3, (c) WC-10Co-10Al_2O_3, and (d) WC-5Co-15Al_2O_3 cermet coatings

Therefore, at high temperature (above 570 ^{0}C) the parameters of oxide layer build-up change rapidly. Namely, a new phase appears, which is FeO. It is believed that the diffusion of Fe^{2+} cations predominates in the iron(II) oxide layer. Moreover, Fe^{2+} and Fe^{3+} cations diffuse in the Fe_3O_4 layer. It turned out that in the Fe_2O_3 phase the transport of oxygen ions in the O^{2-} form predominates [20].

It should be supposed that the diffusion of iron cations towards the oxide layer breaks the structure of WC-Co-Al_2O_3 layer on the C45 substrate, which causes a significant deterioration of the anti-corrosive properties of WC-Co-Al_2O_3 coatings in a hot air environment..

4. CONCLUSIONS

On the basis of the studies were found that:

1. Electrospark deposition (ESD) is a promising process to produce hard and wear-resisting coatings on metallic substrates. Since metallurgical bonded coatings are obtained using ESD technology.
2. The chemical reactions at the interface lead to the formation of various compounds that in turn influence the properties of WC-Co-Al_2O_3 cermet coatings.
3. The microhardness of WC-Co-Al_2O_3 coatings gradually increased with the increase in Al_2O_3 content.
4. The oxidation mechanism of C45 mild steel in an atmosphere of hot air can be considered as electrochemical process. The multi-layer oxide: Fe_2O_3 / Fe_3O_4 / FeO was formed on the substrate.
5. For the tested coatings with increasing of Al_2O_3 content the chemical corrosion rate significantly decreased in the relation to the mild steel without coating. The WC-Co-Al_2O_3 coatings effectively protects of the C45 mild steel surface against chemical corrosion process in an atmosphere of hot air.
6. The oxidation of C45 mild steel covered with WC-Co-Al_2O_3 coatings runs in accordance to the linear law. Therefore, the oxide layer is cracked or porous, and does not protect the substrate against chemical corrosion in high temperature conditions.
7. Heat treatment of the tested materials at 400 ^{0}C improves the condition and appearance of WC-Co-Al_2O_3 cermet coatings.
8. Heat treatment at 800 ^{0}C damages the structure of WC-Co-Al_2O_3 coatings, and thus the anti-corrosion properties of the CC in the hot air atmosphere significantly deteriorate.

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